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RESEARCH INTO THE PREVENTION OF POLYCHLOROTRIPHENYLS (PCTs)

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RESEARCH INTO THE PREVENTION OF POLYCHLOROTRIPHENYLS (PCTs)*

by

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Introduction

In contrast to polychlorobiphenyls (PCBs), on whose subject much research has already been conducted and which we know to be present in the environment as persistent materials, polychlorotriphenyls (PCTs), on the other hand, have seldom been studied. This report presents an inventory of PCT taken, one test sample at a time, in: 1) Rhine water, 2) oysters, 3) eels, and 4) human fat.

Preliminary Processing of the Samples

The four types of samples undergo various types of preliminary processing:

1. Rhine Water

An extract was made of 250 ml water (including silt) with 100, 50, and 50 [sic] ml petroleum ether (40-60). The extract was dried on red-hot $NaSO_4$, filtered and evaporated down to 1 ml. The evaporated extract was submitted to a microcolumn of 2 g Al_2O_3-10% water (Woelm, basic; heated for 16 h at 150°C; after cooling, 90 g Al_2O_3 were mixed with 10 ml water). This was elutriated with 12 ml hexane (Merck, of Rückstand). The elutriated product was evaporated down to a few milliliters.

2. Eels

Of the edible portion of the eel, 25 g were extracted for 8 hours with hexane in a Soxhlet apparatus. The extract was brought up to an exact 25 ml, and the fat content of 5 ml of this solution was determined through evaporation and weighing. Of this solution, 7.5 ml (corresponding to 0.549 g of fat) were stirred together with 10 g of unactivated Florisil, until the solvent evaporated. This mixture was added to 4 g Florisil, also unactivated, and placed in a chromatography tube.

This was elutriated with 70 ml of a 90/10 mixture of acetonitryl and water. The elutriated product was poured into a tap funnel containing 100 ml petroleum ether. After shaking, 300 ml water and 20 ml of a saturated solution of NaCl were added, and the product shaken again. The water stage was then eliminated, and the organic stage was washed two more times, using 50 ml water each time and then letting it evaporate down to a few milliliters.

Afterward, the evaporated and elutriated product was again chromatographed over Al_2O_3, as described in Section 1 for the water extract.

3. Oysters

Extraction and first chromatography were conducted as described in Section 2 for the eels. Second chromatography, however, was not performed over Al_2O_3, but rather over 20 g of re-activated Florisil (reactivation was accomplished through heating at 130°C for one night). Elutriation with 75 ml petroleum ether followed, and the product was evaporated down to a few milliliters.

4. Human Fat

Because of the high fat content of the tissue, the fat could be obtained by direct melting at 90°C; 4 g of the rendered fat were dissolved in 50 ml hexane; 1 ml, corresponding to 80 mg of fat, was chromatographed over Al_2O_3, as described in Section 1.

Research Method

Mass fragmentography is used in analyzing PCT in the various samples. This means that the sample is processed through a combination mass spectrometer / gas chromatograph, with the mass spectrometer set at a characteristic m/e amount, which enables it to function as a specific detector.

For the PCT measurements, the mass spectrometer is set at the following m/e amounts:

m/e = 400 $C_{18}H_9{}^{35}Cl_5$

m/e = 436 $C_{18}H_8{}^{35}Cl_5{}^{37}Cl$ (= $C_{18}H_8Cl_6$)

m/e = 470 $C_{18}H_7{}^{35}Cl_6{}^{37}Cl$ (= $C_{18}H_7Cl_7$)

The value m/e = 400 seemed to have been disturbed, as did the neighboring isotopes. The reason remains unknown at this time. No use is made of this peak for the measurements. The values m/e = 436 and m/e = 470 showed no interference in a blank experiment. m/e = 436 could eventually still receive a contribution from a triphenyl chlorinated eight times and having lost 2 Cl atoms in the ionization process. The mass spectrum made it look as if this was not a significant contribution. Furthermore, as far as measurements are concerned, this is only significant insofar as certain "isomers" are actually triphenyls chlorinated eight times.

Gas-chromatographic conditions for the PCT measurements were as follows:

column	1/8" diam, length 3', pyrex, #3
column filling	3% OV-17 Varaport 30, 80/100 mesh
carrier gas	helium, 24.5-25 ml/min
temperature of injector	310°C
temperature of oven	m/e = 436: 200-285°C, 6°C/min programming m/e = 470: 200-285°C, 8°C/min programming
temperature of detector	310°C
temperature of coupling unit	310°C
temperature of separator	300°C
temperature of line of sight	220°C
temperature of source	220°C
pressure of mass spectrometer	3×10^{-6} Torr

For the retention times of the various isomers and substances, see the pertinent mass fragmentograms (Figs. 1 and 2).

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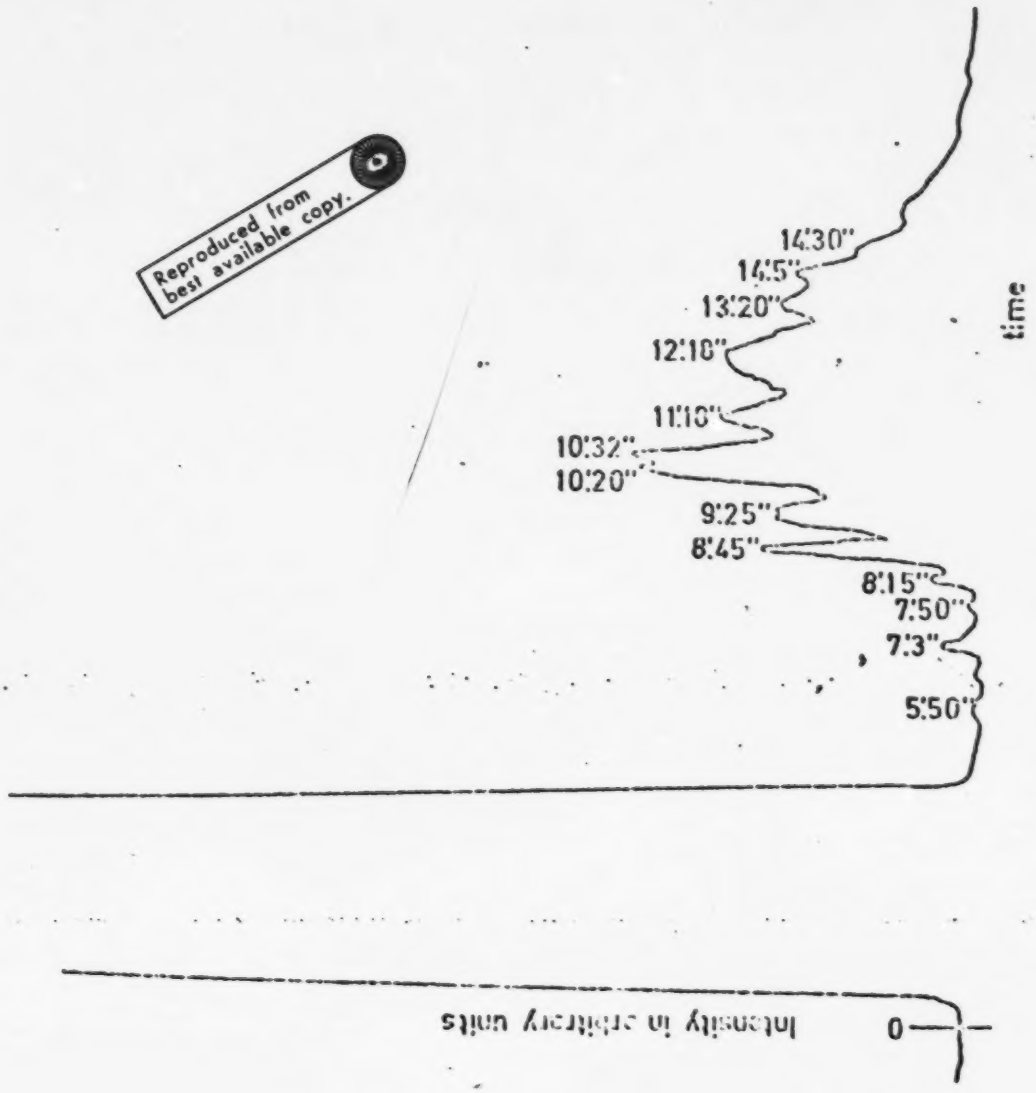

Fig. 1.

Standard Clophen-Harz PCT scale (150 ng)

Sensitivity: Cary 100 mV - BBE 1 V

Set on mass m/e = 436 ($C_{18}H_8{}^{35}Cl_5{}^{37}Cl$)

Temperature of oven: program 6°/min (200→285)

Column: 3' 1/8" 3% OV 17 on Varaport 30

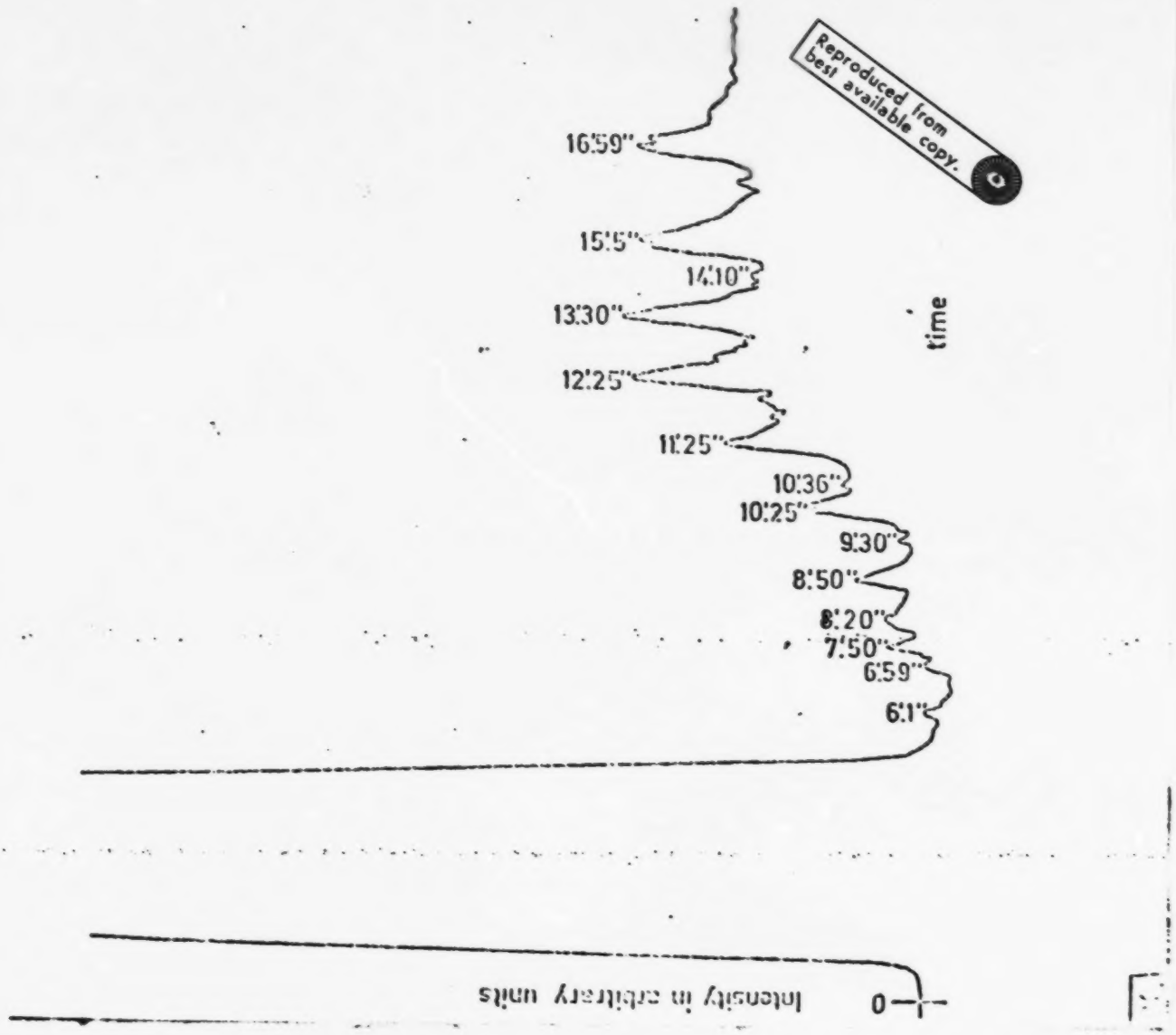

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Fig. 2.

Sample material: oyster (B - 516)

Sensitivity: Cary 30 mV - BBE 0.1 V

Set on mass m/e = 436 ($C_{18}H_8{}^{35}Cl_5{}^{37}Cl$)

Temperature of oven: program 6°/min (200→285)

Column: 3' 1/8" 3% OV17 on Varaport 30

Results

The PCT amounts obtained are always on a standard Clophen-Harz scale.

Averaging is done on two peaks per mass fragmentogram. For m/e = 436, these were the isomers with 8'.50" and 10'.20" retention times. For m/e = 470, they were the isomers with 8'.50" and 9'.48" retention times.

1. Rhine Water

Mixed sample including the following samples: C 1493, C 1624, C 1664, C 1745, and C 1826

m/e = 436	0.08 ppb PCT
m/e = 470	0.05 ppb PCT
Averaged PCT content	0.07 ppb
In comparison, PCB content	0.08 ppb, on basis of Aroclor 1245

2. Eels

Sample B 4374

m/e = 436	0.5 ppm PCT
m/e = 470	0.2 ppm PCT
Averaged PCT content	0.4 ppm
In comparison, PCB content	4.7 ppm, on basis of Aroclor 1254

3. Oysters

Sample B 516

m/e = 436	0.15 ppm PCT
m/e = 470	0.08 ppm PCT
Averaged PCT content	0.12 ppm
In comparison, PCB content	0.2 ppm, on basis of Aroclor 1254

4. Human Fat

Samples B 633, B 638, and B 645, consisting of fat from 1969 corpses;

sample C 1896, consisting of fat of recent date.

B 633	
m/e = 436	PCT not noticeable
m/e = 470	
In comparison, PCB content	0.5 ppm, based on Aroclor 1254
B 638	
m/e = 436	0.5 ppm PCT
m/e = 470	0.5 ppm PCT
Averaged PCT content	0.5 ppm
In comparison, PCB content	0.4 ppm, based on Aroclor 1254
B 645	
m/e = 436	PCT not noticeable
m/e = 470	
In comparison, PCB content:	1.0 ppm, based on Aroclor 1254
C 1896	
m/e = 436	1 ppm PCT
m/e = 470	0.5 ppm PCT
Averaged PCT content	0.8 ppm
In comparison, PCB content:	0.8 ppm, based on Aroclor 1254

Summary and Conclusion

In all samples examined (Rhine water, eels, oysters, and human fat), PCT was found, except in the fat of some corpses dating back to 1969.

The amounts of PCT are somewhat smaller than, but comparable to, the amounts of PCB found. Just like PCB, PCT seems to accumulate in fatty tissue.